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KOKOKU PATENT APPLICATION NO. HEI 7-81114

**A PHOTO-CURABLE ADHESIVE**

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A PHOTO-CURABLE ADHESIVE

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<sup>GW</sup>  
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[p. 2]

(54) [Title of the invention]

A photocurable adhesive

**[Claim of the invention]**

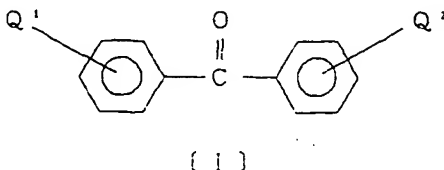
[Claim 1] A photocurable adhesive consisting of component A, component B and component C below.

A: radically polymerizable vinyl compound

B:  $\alpha$ -diketone

C: sensitizer represented by general formula (I).

General formula I



(Wherein, group Q<sup>1</sup> and group Q<sup>2</sup> each independently represents a dialkyl amino group bonded at the o, m, or p position and they can be the same or different.)

**[Detailed description of the invention]**

(a) Objective of the present invention

[Field of industrial application]

The present invention pertains to a photocurable adhesive used for adhesion, sealing or fixing of engaging members of metal components, ceramic components and plastic components.

[Prior art]

The photocurable adhesive is produced by mixing a photopolymerization catalyst with a composition mainly comprising a radically polymerizable vinyl compound, and curing does not take place while shielded from light and the material remains stable for a long time, but the composition easily undergoes polymerization curing upon application of light such as ultraviolet.

Rapid curing upon application of light, especially, low-energy light, is required for the above-mentioned photocurable adhesive.

Conventional photocurable adhesives are used for adhesion of a transparent materials having good light transmittance such as glass, in many cases, and in the case adhesion of semi-transparent materials such as a plastic material containing a ceramic material, pigment, or a filler such as alumina and silica, the energy of light that passes through the material is not sufficient and the dosage is low; furthermore, the sensitivity of the adhesive to the light is not adequate; thus, the composition cannot be used in practice.

[p. 3]

Based on the above background, when adhesion of a semi-transparent material is required, an epoxy-type adhesive, SGA, etc. is used, but mixing of two compositions and a heat treatment at approximately 100°C are required when epoxy-type adhesives are used. On the other hand, when SGA is used, curing can be done at room temperature, but mixing of two compositions is still required, and in order to achieve adequate adhesion, approximately 1 hour of aging is required. As explained above, conventional adhesives capable of bonding semitransparent materials are accompanied by inconvenience and slow curing rate.

[Problems to be solved by the invention]

The objective of the present invention is to produce a fast-curing photocurable adhesive having adequate adhesive strength for transparent materials as well as semitransparent materials without required mixing of two compositions, a high-temperature heat treatment, or an extended aging time, and that is easy to use through achieving an increase in the sensitivity of the adhesive to light.

(b) Constitution of the invention

[Problems to be solved by the invention]

As a result of much research carried out by the present inventors in an effort to eliminate the above-mentioned existing problems, it was found that an adhesive having very high sensitivity to light can be produced when a specific photopolymerization initiator and sensitizer are used, and the present invention was accomplished.

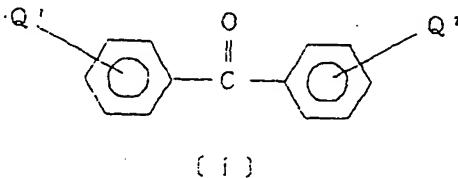
In other words, the present invention is a photocurable adhesive comprising component A, component B and component C shown below.

A: radically polymerizable vinyl compound

B:  $\alpha$ -diketone

C: sensitizer represented by general formula (I).

General formula I



(Wherein, group Q<sup>1</sup> and group Q<sup>2</sup> each independently represents a dialkyl amino group bonded at the o, m, or p position and they can be the same or different.)

In the following, each component that comprises the structure of the adhesive composition of the present invention is explained in further detail.

In the specification of the present invention, the phrase "acrylate and/or methacrylate" is indicated by (meth)acrylate, and the phrase "acrylic acid and/or methacrylic acid" is indicated by (meth)acrylic acid.

[p. 4]

#### <Radically polymerizable vinyl compound>

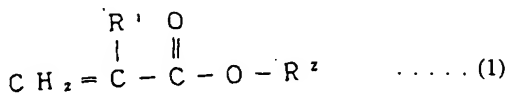
The radically polymerizable vinyl compound used as component A of the adhesive in the present invention is not especially limited as long as it is a compound having a radically polymerizable vinyl group in the molecule.

For specific examples of radically polymerizable vinyl compounds, those listed below can be mentioned.

#### [Vinyl compound (1)]

The compound is a (meth)acrylate represented by following General formula (1).

#### General formula (1)

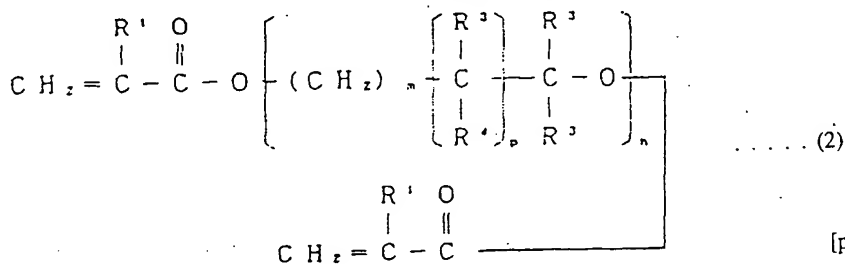


Wherein,  $R^1$  is a hydrogen or  $-CH_3$ ,  $R^2$  is a substituted alkyl group wherein one or two or more hydrogen atoms are replaced with  $-OH$  groups and/or halogens in a straight-chain or branched-chain alkyl group with 2~4 carbon atoms.

For desirable specific examples of the (meth)acrylates represented by general formula (1) above, 2-hydroxyethyl-(meth)acrylate, 2-hydroxypropyl-(meth)acrylate, 1,2-dihydroxyethyl-(meth)acrylate, 3-chloro-2-hydroxypropyl-(meth)acrylate, etc. can be mentioned.

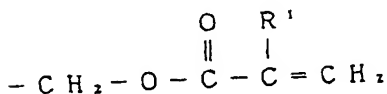
[Vinyl compound (2)]

The compound is a (meth)acrylate represented by general formula (2).

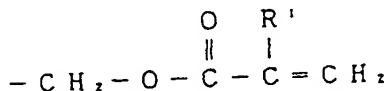


[p. 5]

Wherein,  $R^3$  is a hydrogen,  $-CH_3$ ,  $-C_2H_5$ ,  $-CH_2OH$  or

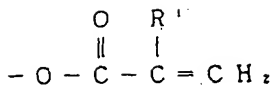


and  $R^4$  is a hydrogen,  $OH$ , or





of

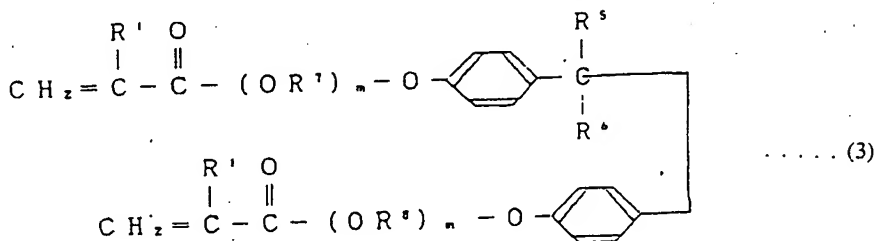


m is an integer in the range of 1 ~ 8, n is an integer in the range of 1 ~ 20, and p is either 0 or 1.

As desirable specific examples of (meth)acrylates represented by general formula (2) above, diethylene glycol di(meth)acrylate, triethylene glycol di(meth)acrylate, tetraethylene glycol di(meth)acrylate, 1,2-propylene glycol di(meth)acrylate, dipropylene glycol di(meth)acrylate, polyethylene glycol di(meth)acrylate, diglycerol di(meth)acrylate, glycerol tri(meth)acrylate, trimethylol propane tri(meth)acrylate, diglycerol tetra(meth)acrylate, etc., can be mentioned.

[Vinyl compound (3)]

The compound is a (meth)acrylate represented by general formula (3).



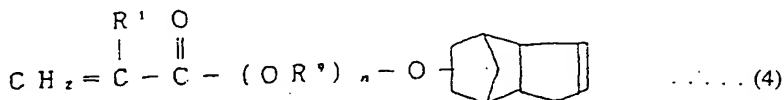
Wherein, R<sup>5</sup> and R<sup>6</sup> are hydrogens or alkyl groups with C<sub>1</sub>~C<sub>8</sub>, R<sup>7</sup> and R<sup>8</sup> are hydrocarbon groups with C<sub>2</sub>~C<sub>8</sub>, and m is an integer in the range of 1~10.

[p. 6]

As desirable specific examples of (meth)acrylates represented by general formula (3) above, 2,2-bis(4-methacryloxy diethoxy phenyl) propane, 2,2-bis(4-allyloxy diethoxy phenyl) propane, 2,2-bis(4-methacryloxy-triethoxy-phenyl) propane, 2,2-bis(4-acryloxy-pentaethoxy-phenyl) propane, 2,2-bis(4-methacryloxy-hexaethoxy-phenyl) propane, 2,2-bis(4-acryloxy heptaethoxy-phenyl) propane, 2,2-bis(4-methacryloxy-octaethoxy phenyl) propane, 2,3-bis(4-acryloxy-dipropoxy phenyl) propane, 2,2-bis(4-methacryloxy-tripropoxy-phenyl) propane, 2,2-bis(4-acryloxy-dibutoxy-phenyl) propane, 2,2-bis(4-methacryloxy octabutoxy-phenyl) propane, 2,2-bis(4-methacryloxy-diethoxy phenyl)-2-(4-methacryloxy-triethoxy-phenyl) propane, 2-(4-acryloxy dipropoxy phenyl)-2-(4-acryloxy-triethoxy-phenyl) propane, etc. can be mentioned.

[Vinyl compound (4)]

The compound is a (meth)acrylate represented by general formula (4).

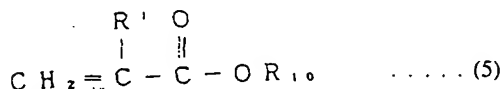


Wherein, R<sup>9</sup> is a hydrogen or hydrocarbon group with C<sub>1</sub> ~ C<sub>4</sub>, and n is an integer in the range of 1 ~ 10.

As desirable specific examples of (meth)acrylates represented by general formula (4) above, dicyclopentenyl (meth)acrylate, dicyclopentenyl methoxy methyl (meth)acrylate, dicyclopentenyl ethoxy ethyl (meth)acrylate.

## [Vinyl compound (5)]

The compound is a (meth)acrylate represented by general formula (5) below.



Wherein, R<sup>10</sup> is a hydrogen, alkyl group with C<sub>1</sub> ~ C<sub>10</sub> or C<sub>5</sub> ~ C<sub>20</sub> cycloalkyl group, phenyl group, tetrahydrofurfuryl group or alkyl group with C<sub>5</sub> ~ C<sub>20</sub> containing those groups.

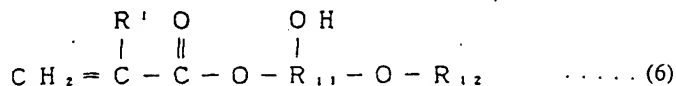
[p. 7]

As desirable specific examples of (meth)acrylates represented by general formula (5) above, (meth)acrylic acid, methyl (meth)acrylate, cyclohexyl (meth)acrylate, tetrahydrofurfuryl (meth)acrylate, 2-ethylhexyl (meth)acrylate, phenyl (meth)acrylate, benzyl (meth)acrylate, etc. can be mentioned.

[p. 7]

## [Vinyl compound (6)]

The compound is a (meth)acrylate represented by general formula (6) below.

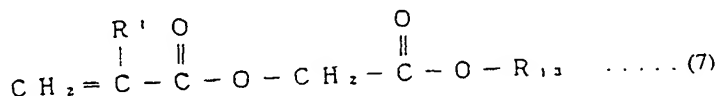


Wherein,  $R^{11}$  is a hydrocarbon group with  $C_2-C_{18}$ , and  $R^{12}$  is a hydrocarbon group with  $C_1-C_{18}$ .

As desirable specific examples of (meth)acrylates represented by general formula (6) above, 2-hydroxy-3-phenoxy propyl (meth)acrylate, cyclohexanone- $\beta$ -hydroxy propyl (meth)acrylate, tetrahydrofurfuryl  $\beta$ -hydroxy propyl (meth)acrylate, and nonyloxy- $\beta$ -hydroxy propyl (meth)acrylate, etc. can be mentioned.

[Vinyl compound (7)]

The compound is a (meth)acrylate represented by general formula (7).



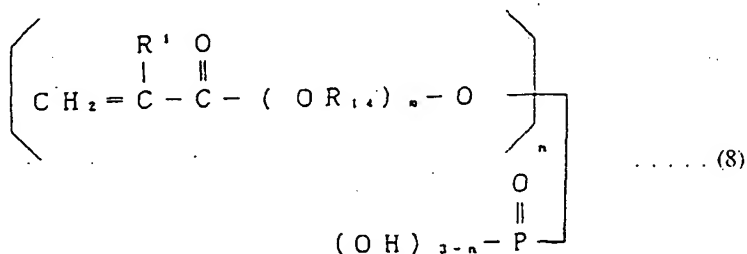
Wherein,  $R^{12}$  is an alkyl group with  $C_1-C_{20}$ , vinyl group, aryl group, or alkoxy alkyl group.

As desirable specific examples of (meth)acrylates represented by general formula (7) above, methoxy carbonyl methyl (meth)acrylate, ethoxy carbonyl methyl (meth)acrylate, heptoxy carbonyl methyl (meth)acrylate, isopropoxy carbonyl methyl (meth)acrylate, etc. can be mentioned.

[p. 8]

[Vinyl compound (8)]

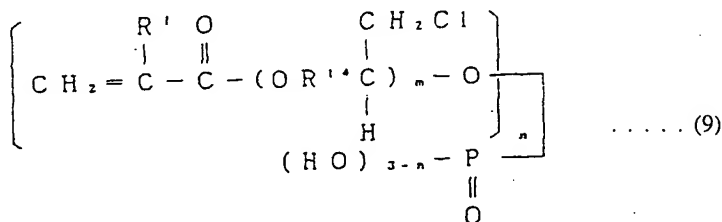
The compound is a (meth)acrylate represented by general formula (8) below.



Wherein,  $\text{R}^{14}$  is a hydrocarbon group with  $\text{C}_1 - \text{C}_{20}$ ,  $m$  is an integer in the range of 1 ~ 10, and  $n$  is either 1 or 2.

[Vinyl compound (9)]

The compound is a (meth)acrylate represented by general formula (9).



Wherein,  $m$  is an integer in the range of 1 ~ 10, and  $n$  is either 1 or 2.

As desirable specific examples of (meth)acrylates represented by general formula (8) above and general formula (9), acid phosphoxy ethyl (meth)acrylate, 3-chloro-2-acid phosphoxy propyl (meth)acrylate, and phosphoxy propyl(meth)acrylate acid, etc. can be mentioned.

[Other radically polymerizable vinyl compounds]

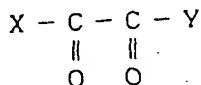
In addition to the above-mentioned radically polymerizable vinyl compounds, vinyl esters such as isobonyl (meth)acrylate, hydrogenated pentadienyl (meth)acrylate, vinyl acetate, and vinyl propionate, vinyl ethers such as methyl vinyl ether, ethyl vinyl ether, and cyclohexyl vinyl ether, alkenyl benzenes such as styrene, vinyl toluene,  $\alpha$ -methyl styrene, and chloromethyl styrene, and furthermore, N-vinyl pyrrolidone, etc. can be mentioned as component A of the present invention.

The above-mentioned radically polymerizable vinyl compounds can be used independently or two, or more different types of the above-mentioned compounds can be used in combination, as component A of the present invention.

[p. 9]

#### < $\alpha$ -diketone>

$\alpha$ -diketone of component B in the adhesive of the present invention absorbs light in the near-ultraviolet or visible region and acts as a photopolymerization initiator, and those represented by general formula (II) are especially desirable.



(Wherein, X and Y are substituted or non-substituted alkyl groups, alkenyl groups or aryl groups, and X and Y may be bonded and form a ring structure.)

As desirable specific examples of  $\alpha$ -diketones represented by the general formula above, camphorquinone, benzyl,  $\alpha$ -naphthyl, acetophenone, p,p-dimethoxy benzyl, p,p'-dichlorobenzyl-diacetyl, butane dione, pentane dione, 1,2-phenanthrene quinone, 1,4-

phenanthrene quinone, 3,4-phenanthrene quinone, 9,10-phenanthrene quinone, naphthoquinone, etc. can be mentioned, and as an especially desirable  $\alpha$ -diketones, in this case, camphoquinone, benzyl, and butane dione can be mentioned.

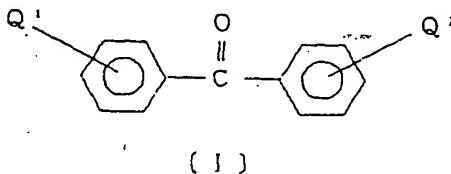
The above-mentioned  $\alpha$ -diketones can be used independently, or two or more different types of the above-mentioned compounds can be used in combination, as component B of the present invention.

The mixing ratio of component B varies depending on the radically polymerizable vinyl compound used but it is a theoretical catalytic amount capable of providing the function of a photopolymerization catalyst and capable of imparting adequate photocurable properties to the composition, and in general, 0.01 ~ 20 parts by weight (hereinafter parts) for 100 parts of component A, and in the range of 0.05 ~ 10 parts is further desirable.

< Sensitizer >

Component C used in combination with the above-mentioned  $\alpha$ -diketone as a sensitizer in the present invention is represented by general formula (I) below.

General formula I



(Wherein, group  $Q^1$  and group  $Q^2$  each independently represents a dialkylamino group bonded at the o, m, or p position and they may be the same or different.)

[p. 10]

In the above-mentioned general formula (I), a desirable dialkylamino group is a lower dialkylamino group such as a dimethylamino group, diethylamino group, dipropylamino group, dibutylamino group, and methylethylamino group, and as desirable specific examples of component C, 4,4'-bis(diethyl-amino)benzophenone, 2,4'-bis(diethylamino) benzophenone, 3,4'-bis(diethylamino) benzophenone, 2,2'-bis(diethylamino) benzophenone, 2,3'-bis(diethylamino) benzophenone, 4,4'-bis(dimethylamino) benzophenone, 2,2'-bis(dimethyl amino) benzophenone, 2,4'-bis(dimethyl amino) benzophenone, 4,4'-bis(dipropylamino) benzophenone, 2,4'-bis(dipropylamino) benzophenone, 4,4'-bis(dibutylamino) benzophenone, 2,4'-bis(dibutylamino) benzophenone, 4-methylethylamino-4'-dimethylamino benzophenone, and 4-methylethylamino-2'-dimethylamino benzophenone, etc. can be mentioned.

The behavior of the sensitizer (component C) in the present invention at the time of polymerization reaction is not well understood at this point, but it is hypothesized that the sensitizer absorbs light in the ultraviolet-visible region and promotes decomposition of the  $\alpha$ -diketone generated and formation of a radical.

One or a mixture of two or more different types of amine compounds having the structure shown above in general formula (I) are used as the sensitizer.

The mixing ratio of component C varies depending on the radically polymerizable vinyl compound used but it is a catalytically theoretical amount capable of achieving the function of the photopolymerization catalyst and capable of imparting adequate photocurable properties to the composition, and in general, 0.01 ~ 20 parts for 100 parts of component A, and in the range of 0.05 ~ 10 parts is further desirable.

< Optional components >



In addition to the above-mentioned components of A, B and C, a variety of substances can be included for different purposes. For example, nitro compounds, nitroso compounds, and quinones can be included to increase stability, dyes and pigments can be included for coloring, thixotropic agents such as silica can be included to impart thixotropic properties, acrylic resins, urethane resins, epoxy resins, silica, etc. can be included for increasing the viscosity or weight.

Furthermore, an organic peroxide can be included in the adhesive of the present invention to impart thermopolymeric properties or anaerobic polymeric properties.

Production of the adhesive composition of the present invention can be easily achieved upon mixing the above-mentioned each components at ambient temperature or under heat.

[p. 11]

<Adhesion method>

In order to perform bonding using the adhesive of the present invention, a coating is applied to one transparent or semitransparent material and the object to be bonded is lightly pressed against the material and an effective dosage of ultraviolet or visible light is applied to the object being bonded.

The dosage of light is not especially limited, so long as an effective dosage is applied, and standard light sources can be used in this case, and those listed below can be used effectively: carbon arc lamp, mercury steam arc lamp, fluorescence lamp, argon glow lamp, white incandescent lamp, halogen lamp, high-pressure mercury lamp, ultra-high-pressure mercury lamp, low-pressure mercury lamp, deep-UV lamp, xenon short lamp, high output

water cooled xenon lamp, xenon flash lamp, gallium halide lamp, thallium halide lamp, metal halide lamp, etc.

[Application examples and comparative examples]

In the following, the present invention is further explained in specific terms with application examples and comparative examples but the present invention is not limited to these examples.

The outer area of the mixing container was covered with aluminum foil, and for 100 parts of a specific radically polymerizable vinyl compound, a specific photopolymerization catalyst and sensitizer were added to the mix, and stirring was performed at a heat treatment temperature of 80°C to produce a homogeneous solution, and a photocurable adhesive with the composition shown in Table I was produced.

It should be noted that 2-hydroxy-2-methyl propiophenone used for production of the Comparison Adhesive No. 1 is a material commonly used as an ultraviolet-curable polymerization initiator.

Table I: Photocurable adhesives

|   |   | Photocurable adhesive (parts by weight) |     |     |     |     |     |     |     |     |     |     |     | Comparative Example No. |     |     |
|---|---|---|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-------------------------|-----|-----|
|   |   | Application Example No.                 |     |     |     |     |     |     |     |     |     |     |     |                         |     |     |
|   |   | 1                                       | 2   | 3   | 4   | 5   | 6   | 7   | 8   | 9   | 10  | 11  | 12  | 1                       | 2   | 3   |
| A | 2,2-bis(4-acryloxy diethoxy phenyl) propane | 100                                     | 0   | 0   | 0   | 0   | 0   | 0   | 0   | 0   | 0   | 0   | 0   | 100                     | 0   |     |
|   | Trimethylol propane triacrylate             | 0                                       | 100 | 0   | 0   | 0   | 0   | 0   | 100 | 100 | 100 | 0   | 0   | 0                       | 100 | 0   |
|   | Hydrogenated dicyclopentadienyl diacrylate  | 0                                       | 0   | 100 | 0   | 0   | 0   | 0   | 0   | 0   | 0   | 0   | 0   | 0                       | 0   | 0   |
|   | Tetraethylene glycol diacrylate             | 0                                       | 0   | 0   | 100 | 0   | 0   | 100 | 0   | 0   | 0   | 100 | 100 | 0                       | 0   | 100 |
|   | Acrylic oligomer M8080                      | 0                                       | 0   | 0   | 0   | 100 | 0   | 0   | 0   | 0   | 0   | 0   | 0   | 0                       | 0   | 0   |
|   | 2-hydroxy-3-phenoxy propyl acrylate         | 0                                       | 0   | 0   | 0   | 0   | 100 | 0   | 0   | 0   | 0   | 0   | 0   | 0                       | 0   | 0   |
| B | Camphorquinone                              | 0.4                                     | 0.4 | 0.4 | 0.4 | 0.4 | 0.4 | 1   | 1   | 0   | 0   | 0   | 0   | 0                       | 0   | 0   |
|   | Benzyl                                      | 0                                       | 0   | 0   | 0   | 0   | 0   | 0   | 0   | 1   | 2   | 0.4 | 0.4 | 0                       | 1   | 1   |
| C | 4,4'-bis(dimethylamino) benzophenone        | 0.1                                     | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 | 0   | 0   | 0.2 | 0.2 | 0   | 0.2 | 0                       | 0   | 0   |
|   | 4,4'-bis(diethylamino) benzophenone         | 0                                       | 0   | 0   | 0   | 0   | 0   | 0.4 | 0.4 | 0   | 0   | 0.4 | 0   | 0                       | 0   | 0   |
| — | 2-hydroxy-2-methylpropiofenone              | 0                                       | 0   | 0   | 0   | 0   | 0   | 0   | 0   | 0   | 0   | 0   | 0   | 1                       | 0   | 0   |

Components

## [Evaluation of photocurable adhesive]

Each photocurable adhesive listed in the above-mentioned Table I was sandwiched between two test pieces shown below and a halogen lamp was applied to the test piece from a distance of 32 mm above the test piece for 60 seconds and curing of the adhesive was performed.

Furthermore, the illumination level of the transmitted light at a specific measuring wavelength when the light from the halogen lamp is applied through the test piece is as shown in the following Table II, and ultraviolet was hardly included in the light that passed through the test piece and a very weak visible light alone was included.

< Test piece >

Trade name: Polybutylene terephthalate

[Product of Nippon Test Panel Ind. (Ltd.)]

Specification: Juranex 2002 (white)

[p. 13]

Dimension: 2.0 mm (thickness) x 25 mm (width) x 100 mm (length)

Table II (luminous intensity of light after passing through the test piece)

| Measurement wavelength (nm) | Luminous intensity (mw/cm <sup>2</sup> ) |
|-----------------------------|--|
| 365                         | Too low, measurement not possible        |
| 405                         | 2.05                                     |
| 436                         | 0.42                                     |

Curing of the adhesive was carried out as described above and measurement of the tensile shear adhesive strength and flexural adhesion were done according to the specifications of JIS K-6850 and JIS K-6856, respectively. The results obtained are shown in Table III and Table IV.

Table III (Results of tensile shear adhesion strength)

| Photocurable adhesive     | Tensile shear adhesive strength (gf/cm <sup>2</sup> ) |
|---------------------------|---|
| Application Example No. 1 | 4,000   |
| Application Example No. 2 | 6,000   |
| Application Example No. 3 | 6,000   |
| Application Example No. 4 | 5,000   |
| Application Example No. 5 | 6,500   |
| Application Example No. 6 | 8,000   |
| Comparative Example No. 1 | 0   |
| Comparative Example No. 2 | 0   |
| Comparative Example No. 3 | 0   |

Table IV (Results of flexural adhesion test)

| Photocurable adhesive      | Flexural adhesion (g) |
|----------------------------|-----------------------|
| Application Example No. 7  | 2,100                 |
| Application Example No. 8  | 710                   |
| Application Example No. 9  | 600                   |
| Application Example No. 10 | 1,100                 |
| Application Example No. 11 | 200                   |
| Application Example No. 12 | 500                   |
| Comparative Example No. 1  | 0                     |
| Comparative Example No. 2  | 0                     |
| Comparative Example No. 3  | 0                     |

[p. 14]

As indicated by the results obtained above in the application examples and comparative examples, adhesive strength was absent in photocurable adhesives mixed with conventional photopolymerization catalysts upon bonding of semi-transparent polybutylene terephthalate in which ultraviolet with a high energy density is hardly transmitted and visible light with a low energy density is only slightly transmitted since a white pigment is included;

on the other hand, excellent photocurability and adhesive strength can be achieved in the photocurable adhesive of the present invention.

[Effect of the invention]

The photocurable adhesive of the present invention has excellent ultraviolet curability as well as harmless visible-light curability and excellent sensitivity, and adhesive strength can be achieved upon bonding transparent materials as well as semitransparent materials, for example, ceramic materials such as alumina and silica, and plastic materials containing a pigment or ceramic filler, where ultraviolet with a high energy density is hardly transmitted and only visible light with a low energy density can be transmitted.